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OAK RIDGE NATIONAL LABORATORY

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U.S. ATOMIC ENERGY COMMISSION

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ABSTRACT

Contamination of the atmosphere by radioactive isotopes of iodine constitutes a serious biological hazard and, for this reason, provisions should be made at reactors to prevent such releases in the event of an accident. The efficiency of activated charcoal for adsorption of iodine vapor from air streams was measured by using a radioactive tracer method, and efficiencies of 99.6 to 99.999+% were obtained for various conditions. Comparative tests were run with silver-plated copper ribbon. A criterion for selecting an iodine removal material was developed based on efficiency and resistance to air flow. The iodine vapor adsorption efficiency of a commercial charcoal filter was measured. Various materials were considered for possible application in the emergency exhaust system of the building housing the 5-Mw swimming pool reactor at the Puerto Rico Nuclear Center. Based upon its high adsorption efficiency and retention properties, it is proposed that activated charcoal be utilized for iodine vapor adsorption.

INTRODUCTION

Radioactive isotopes of iodine, by-products of fissioning U^{235}, present a somewhat unique biological hazard because inhaled and ingested iodine is concentrated in the thyroid gland. For this reason the release of radioactive iodine vapor into the atmosphere should be avoided. Very large quantities of iodine isotopes are contained in the fuel of operating nuclear reactors. As an example, a reactor operating for 39 days at an average flux of 10^{14} neutrons$\cdot$cm$^{-2}\cdot$sec^{-1} will contain approximately 2.5×10^5 curies of mixed isotopes of iodine per megawatt of power level. A reactor accident, such as core meltdown, could release a large fraction of this iodine. Provisions must be made to prevent this iodine vapor from entering the atmosphere and contaminating the surrounding populated areas.

This study was conducted to determine a feasible method for iodine vapor adsorption to be used in the emergency ventilation system of the building housing the 5-Mw swimming pool reactor at the Puerto Rico Nuclear Center (PRNC). In the event of a reactor accident, building air will be diverted from the normal exhaust system and passed through the emergency exhaust system which will contain provisions for the removal of particulate matter in addition to iodine vapor. This system must be capable of going into immediate operation without supervision and must continue to operate remotely until the contaminated building air has been processed and reactor personnel are able to re-enter the building. For contamination control to be successful, the system must remove radioiodine vapor and particulate matter from the exhaust air with high efficiency and contain this material for a length of time greater than that required to completely change the building atmosphere.

METHODS FOR ADSORPTION OF IODINE VAPOR

Various methods have been reported for removal of iodine vapor from air streams. These methods may be grouped into several categories: gas scrubbers utilizing liquid absorbents (*9, 14, 16*); systems based upon the reaction between iodine vapor and silver nitrate at high temperatures (*3, 11, 12*); and systems using solid adsorbents at room temperature (*1, 4, 7, 15, 17*).

The mode of emergency operation in the event of a reactor accident places certain restrictions on the method to be utilized for iodine vapor adsorption. The necessity of a high operating temperature (200°C) precludes the use of a silver-iodine reactor, and the mechanical problems associated with handling and maintaining large volumes of a liquid absorbent in a gas scrubber limit its application in this case. A system employing a solid adsorbent appears to be more easily adapted to the requirements of the PRNC exhaust system.

A survey of the literature reveals that many solid materials have been studied for possible use as iodine adsorbents. Materials such as zinc granules, potassium hydroxide pellets, tin ribbon, silver-plated copper ribbon, activated alumina, porous glass spheres, activated charcoal, copper ribbon, and slag wool fibers coated with potassium oxide, silver nitrate, cadmium, cadmium-antimony, or silver have been tested for iodine vapor adsorption by the Harvard University Air Cleaning Laboratory (*15*). Other materials reported in the literature are Linde molecular sieves, silica gel, calcium hydroxide, and soda lime.

EXPERIMENTAL

A laboratory study of the more promising materials was conducted utilizing the system diagramed in Fig. 1. Wherever possible, the system was constructed of glass to minimize adsorption of the iodine vapor by the walls. A typical experiment involved the following operations. Elemental iodine crystals (I^{127} containing radioactive I^{131}) were contained in the U-tube, and a portion of the air supply was routed through the U-tube to sweep iodine vapor into the main air stream. The time required for introduction of all the iodine vapor into the adsorber column was approximately 15 min, and the average iodine concentration during this time was 0.18 mg of I^{127} and 54 μc of I^{131} per cubic foot of air. Air flow through the system was then continued for 24 hr. Iodine vapor escaping from the adsorber column was collected downstream by the combination of a plug of carbon wool fibers,[1] a CWS-6 absolute filter, and an electrostatic precipitator. After completion of the 24-hr cycle, the apparatus was disassembled and the distribution of iodine radioactivity in the adsorber column was determined by scanning with a sodium iodide scintillation crystal viewing through a small slit in a lead shield. The over-all efficiency of the system was then determined by radiochemical assay of the entire system from point A to point B in Fig. 1. By comparing the amount of iodine residing in the adsorber column with the amount found in the total system, an adsorption efficiency was determined. It was realized that the accuracy of this method for determining iodine adsorption efficiency depends upon the premises that all iodine passing through the adsorber was collected and that none was allowed to escape. All efforts to detect iodine activity in the effluent air were unsuccessful. In calculating the efficiency, all downstream samples (i.e., CWS-6 filter paper, aluminum liner from electrostatic precipitator, etc.) in which radioiodine could not be detected were assumed to contain an amount of I^{131} equal to the

[1]Available from Barnebey-Cheney Co., Columbus, Ohio.

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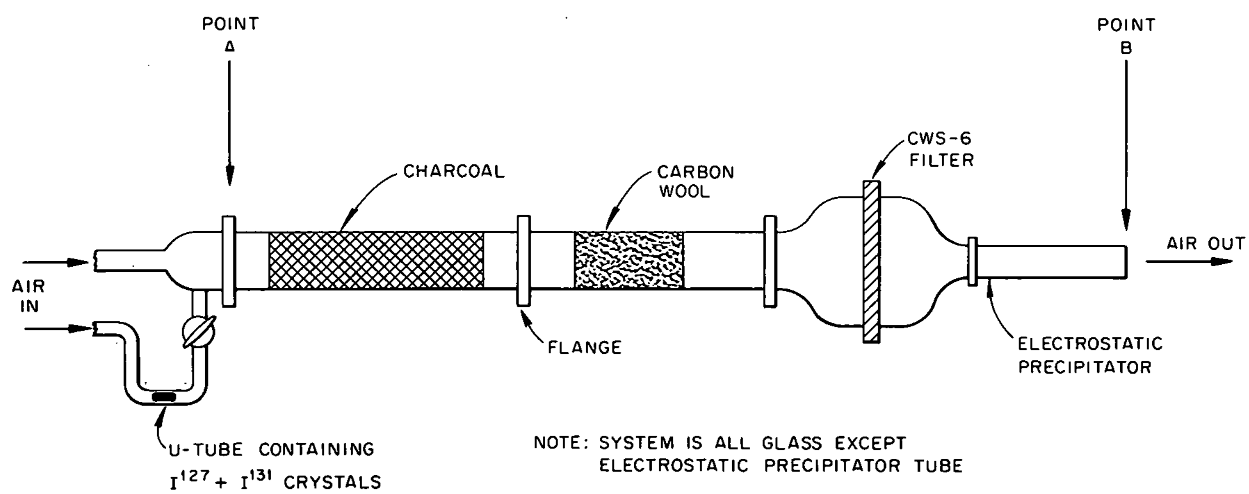

Fig. 1. Experimental System.

limit of detection of the radiochemical method of assay. Therefore, the iodine adsorption efficiency calculated is less than the true efficiency and represents a lower limit.

The iodine tracer was prepared by adding the appropriate amount of I^{127} as NaI to a basic sodium sulfite solution of I^{131}. The iodine was precipitated as palladium iodide, recovered, and dried under vacuum. To recover the tagged iodine, the dry precipitate was decomposed by heating in vacuum, and the liberated iodine vapor was collected in a U-tube cooled by liquid nitrogen. This U-tube then served as the iodine container for the experimental system.

Activated Charcoal

The major portion of this study was centered on charcoal since this material has been shown to have a very high efficiency for iodine vapor adsorption under various conditions. Parameters selected for examination were limited to those of interest in the PRNC application.

For proper design of an iodine adsorber the effect of air velocity and adsorbent particle size on the adsorption process must be known. The effect of superficial air velocity (volumetric air flow divided by cross-sectional area of adsorbent container) on iodine adsorption was studied at velocities of 82, 170, and 275 fpm through adsorbers containing 6–8 mesh charcoal. Very little difference was observed. The depth of penetration into the charcoal mass and the over-all efficiency of the system were almost equal for the three air velocities. The size of the charcoal particles does affect the adsorption efficiency. Study of 2–4, 4–6, and 6–8 mesh charcoal (Columbia SXC) at a superficial air velocity of 170 fpm yielded efficiencies of 99.63, 99.89, and 99.99+%, respectively. A typical iodine distribution for an adsorber 8 in. deep, containing 6–8 mesh Columbia SXC charcoal, and operated at a linear air velocity of 170 fpm is given in Fig. 2. For optimum performance an iodine adsorber should contain charcoal of particle size 6–8 mesh, or smaller, and even though little effect of air velocity was noted over the range studied, the air velocity should be kept low because of pressure drop and prevention of mechanical damage to the system.

Early in the study it was observed that dust particles in the air sweep might be responsible for transport of iodine through an adsorber: iodine adsorbed on a particle of dust would not be available for reaction at the charcoal surface. In addition, fine particles of charcoal containing iodine might be carried from the adsorber by the air sweep. For these reasons the absolute filter (CWS-6) and the electrostatic precipitator were

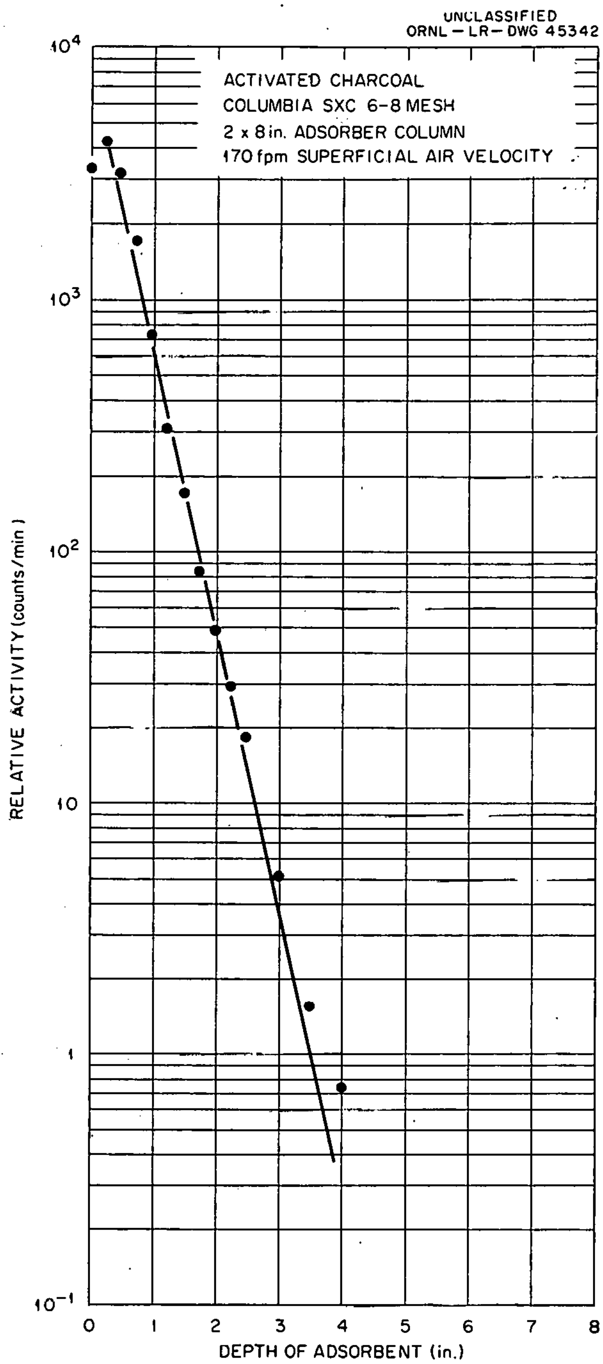

Fig. 2. Distribution of I^{131} Radioactivity in Charcoal Column.

included in the experimental system in addition to the carbon wool. In one run, the CWS-6 filter was placed immediately downstream from the charcoal mass, and particularly "dusty" charcoal was used. A significant amount of dust and iodine activity was found on the filter, and a detectable trace was found on the inner surface of the electrostatic precipitator. In all later runs the dust was removed from the charcoal before iodine injection by introducing air at a flow rate greater than that to be used in the experiment, and the dust problem was greatly reduced.

In the PRNC application, the adsorber will be required to process contaminated air laden with moisture resulting from vaporized pool water. Possible interference by moisture contained in the air and adsorbed on the charcoal with iodine adsorption was investigated. Identical adsorbers were constructed and tested, one with dry air and the other with moist air at 82% relative humidity. The charcoal used in the wet test was exposed to moist air flow until an equilibrium quantity of moisture was contained by the charcoal, prior to the introduction of the iodine vapor. No significant effect was noted, as evidenced by an efficiency of 99.9956 and 99.9936% for the dry and wet test, respectively.

Most of the tests were operated for a 24-hr period, and during this time no downstream transport of iodine could be detected; the major portion of the iodine was concentrated at the inlet of the adsorber. One test was operated for 70 hr after injection of the iodine, and no movement could be detected. Once adsorption takes place, the iodine is firmly held on the charcoal surface. In one reported study (*7*), the flow rate was increased by a factor of 20 and the temperature increased from 25°C to 80°C, but the attempt to cause iodine movement after adsorption had occurred was unsuccessful.

All tests were made at room temperature, since increased temperature had not been noted by other workers to decrease iodine adsorption efficiency. In one case, charcoal at a temperature in excess of 100°C in helium was successfully applied for iodine vapor adsorption (*6*). To prevent excessive oxidation of charcoal by air, the temperature should not be allowed to exceed 100°C for long periods of time.

The iodine adsorption system at PRNC will not be exposed to air flow until called upon to process building air in the event of a reactor emergency. Charcoal would not be expected to lose iodine adsorption efficiency upon exposure to air; however, to check this point, one test was made on charcoal that had been exposed to the laboratory atmosphere for several weeks and then exposed to air flow for 350 hr prior to iodine vapor injection. An efficiency of 99.99+% was obtained, and no detectable effect on iodine vapor adsorption was noted.

Silver-Plated Copper Ribbon

A large part of the work on iodine vapor adsorption at the Harvard University Air Cleaning Laboratory has been centered on silver-plated copper ribbon[2] (*15*). Experiments have been conducted with air sweep at velocities ranging from 60 to 343 fpm, with iodine concentrations ranging from 10^{-3} to 30 mg/ft^3, and at temperatures ranging from 25 to 300°C. It was shown that iodine retention by 25- by 2-mil silver-plated copper ribbon at room temperature ranged up to 99% and that at 300°C the efficiency increased to approximately 99.9%. Once the ribbon was exposed to high temperature, the efficiency dropped to 31% upon cooling to room temperature. Iodine collection efficiency was also noted to be concentration dependent. The efficiency for 25- by 2-mil ribbon, at a packing density of 33 lb/ft^3, was estimated to be 90% for an iodine concentration of 1×10^{-3} mg/ft^3. The efficiency can be increased to approximately 98%, however, if the ribbon dimensions are decreased to 3 by 2 mils and the packing density increased to 71 lb/ft^3.

Three tests were made in this study by employing silver-plated copper ribbon as the adsorbent. The ribbon was 25 mils wide by 2 mils thick, woven into a mesh configuration, and coated with silver equal to 5% by weight. The runs were conducted at a face velocity of 170 fpm and with iodine concentrations of 0.18 mg of I^{127} and 54 μc of I^{131} per cubic foot of air. In the first test, the air supply was filtered through a compressed air filter ("Fulflo") which had been used in the charcoal study. The adsorber contained eight ribbon pads compressed to a packing density of 25 lb/ft^3. The iodine adsorption efficiency of the system was 92%, with an iodine

[2]Available from Metal Textile Co., Roselle, N. J.

distribution in the adsorber as shown in Fig. 3. A break is noted in the distribution curve at a depth of approximately 4 in., indicating that the efficiency beyond this depth is much lower than at the entrance. This effect has also been noted in the work at Harvard. The other two tests were run under the same conditions as before except that a CWS-6 filter was added at the air supply. The adsorption efficiency was increased to 98 and 99%, indicating that particulate matter in the air supply may be responsible for the transport of iodine through the silver-copper ribbon.

Copper Ribbon

One test employing new copper ribbon was made under conditions similar to those of the silver-copper tests. An iodine efficiency of 98.5% was obtained with a distribution as shown in Fig. 4. Tests at Harvard indicate that copper ribbon and silver-copper ribbon have comparable iodine

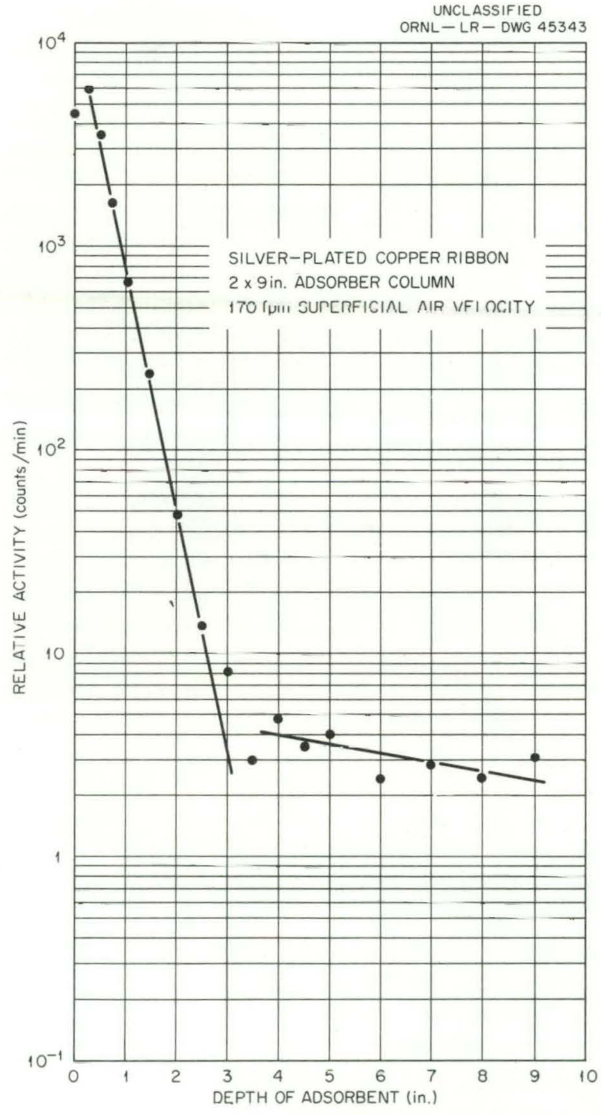

Fig. 3. Distribution of I^{131} Radioactivity in Silver-Plated Copper Ribbon Column.

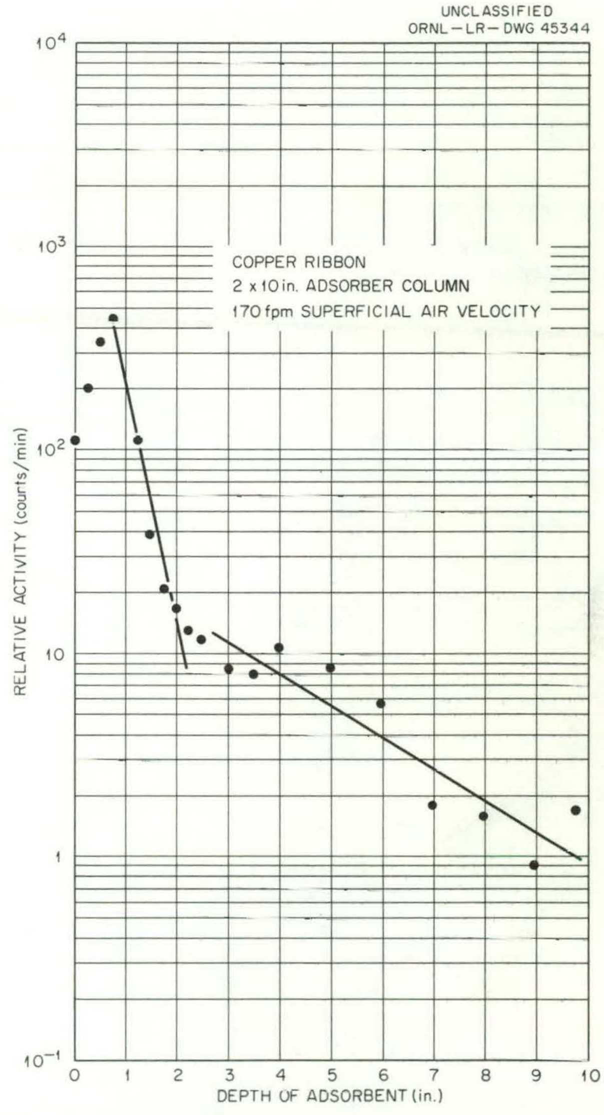

Fig. 4. Distribution of I^{131} Radioactivity in Copper Ribbon Column.

collection properties at room temperature. At elevated temperatures (300°C) the copper ribbon failed after 25 hr, while the silver-copper ribbon showed no indication of reduced efficiency after 100 hr of hot operation (*15*).

COMPARISON OF ACTIVATED CHARCOAL AND SILVER-PLATED COPPER RIBBON

The choice of using activated charcoal or silver-copper ribbon for iodine adsorption will depend upon the application. Each material is subject to shortcomings under various conditions.

Activated charcoal is a more efficient adsorbent, but the pressure drop through the charcoal mass is significant in some applications. Silver-copper ribbon, while being somewhat less efficient, exhibits an almost insignificant pressure drop. Figure 5 displays pressure drop as a function of superficial air velocity for several mesh sizes of charcoal and for silver-copper ribbon at a packing density of 27 lb/ft^3.

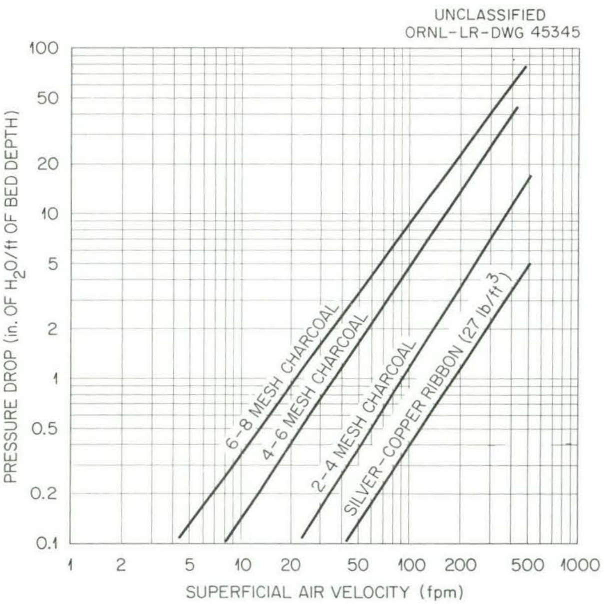

Fig. 5. Pressure Drop vs Superficial Air Velocity.

Upon comparison of the distribution curves of iodine adsorbed by charcoal and silver-copper ribbon a distinct difference is noted. The iodine distribution line for charcoal does not "break," indicating that the adsorption efficiency is constant over the length of the adsorber. The distribution curve for silver-copper ribbon breaks and changes slope at a depth of approximately 4 in. This effect could result from a different mechanism of adsorption becoming predominant at this depth and is thought to indicate that iodine vapor is the species being adsorbed at the entrance to the adsorber while the species giving rise to the break is due to iodine adsorbed on dust particles which are penetrating the adsorber. Efficient prefiltration of the air supply reduced this effect somewhat.

The application of charcoal for iodine adsorption may be limited somewhat by its tendency toward rapid oxidation at high temperatures in an oxidizing atmosphere (*2*). Heating of the charcoal can occur from both the beta decay of adsorbed radioiodine and the heat content of the gas containing the iodine vapor. For this reason, the use of charcoal in systems where the temperature is high (>150°C) and oxidizing gases are present should be viewed cautiously, and some means of cooling the gas stream or charcoal applied. Silver-copper ribbon gains in efficiency when the temperature increases but reaches a maximum at 300°C, and at higher temperatures the collected iodine is released. One factor to be noted is that once exposed to high temperatures the silver-copper ribbon is no longer effective at room temperature.

Silver-copper ribbon exhibits a sensitivity to iodine concentration in the air. At low concentrations (10^{-3} mg/ft^3) the efficiency may drop to 90% as compared with 98 to 99% when the concentration is in the milligrams per cubic foot range. Charcoal has not exhibited this sensitivity and has a high efficiency over a wide concentration range. Earlier work at this Laboratory (*1*) indicated that charcoal has an efficiency of 99.95% at concentrations of approximately 10^{-6} mg of I^{131} per cubic foot of air. One test reported by workers at Harvard gave an efficiency of 96.8% for 8–14 mesh charcoal at an iodine concentration of approximately 4×10^{-9} mg of I^{131} per cubic foot of air.

It seems that the choice of using charcoal or silver-copper ribbon will depend mainly upon the decontamination efficiency desired in the air cleanup and upon the pressure drop that can be tolerated. On the basis of decontamination efficiency alone, 6–8 mesh charcoal is superior to the other adsorbents tested. In Fig. 6 the various mesh sizes of charcoal and silver-copper ribbon are compared, based upon decontamination factor as a function of depth of adsorbent. The multitude

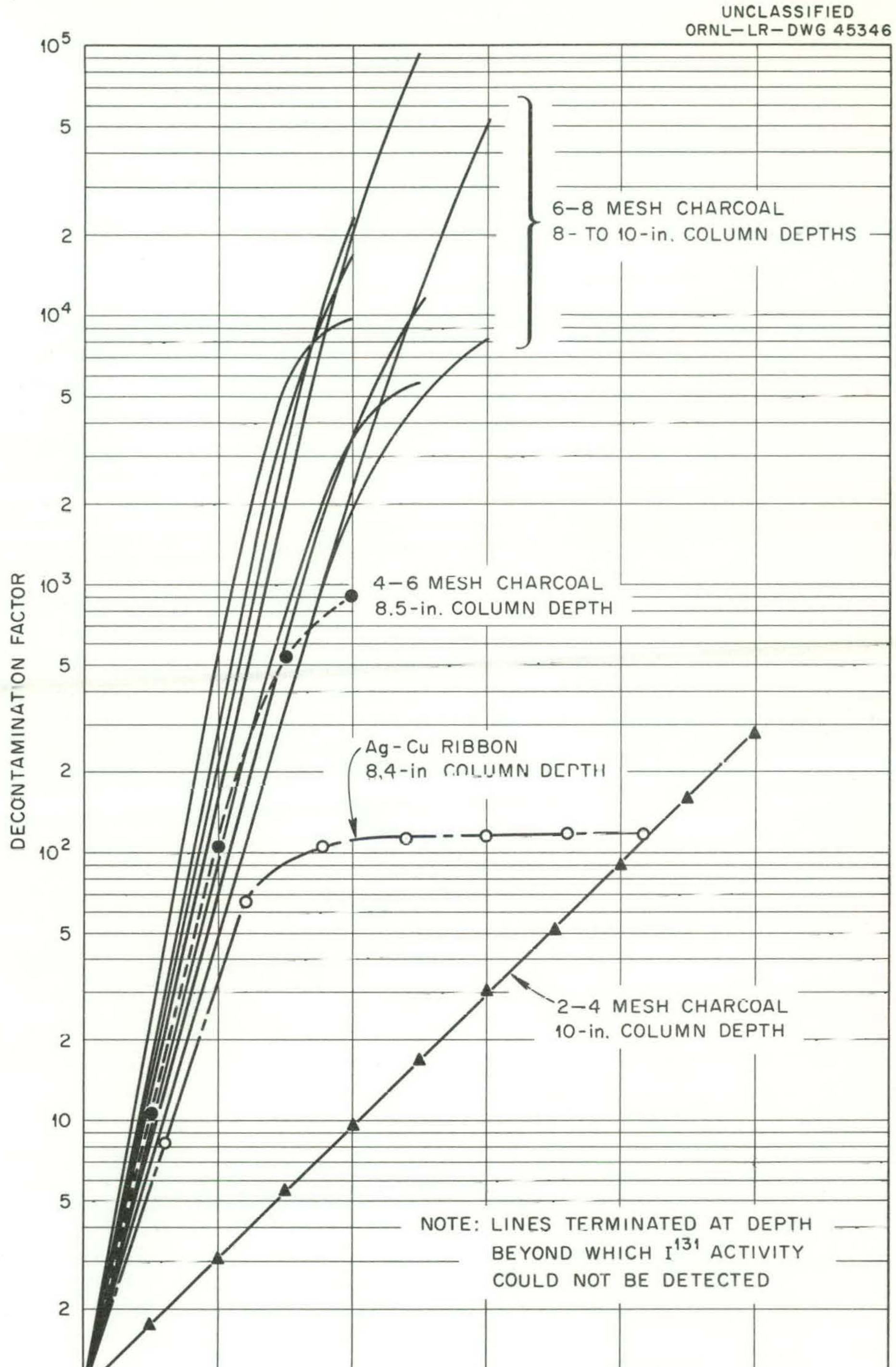

Fig. 6. Decontamination Factor as a Function of Adsorbent Depth.

of lines for 6–8 mesh charcoal represents tests under the various conditions of superficial air velocity, moisture content, and duration of air sweep. The decontamination factor is defined as the amount of iodine found in the adsorber column divided by the amount of iodine which passed through. In an attempt to relate pressure drop, superficial air velocity, and decontamination efficiency, a plot was made of the decontamination factor as a function of pressure drop divided by superficial air velocity. This information is contained in Fig. 7. The curve for 6–8 mesh

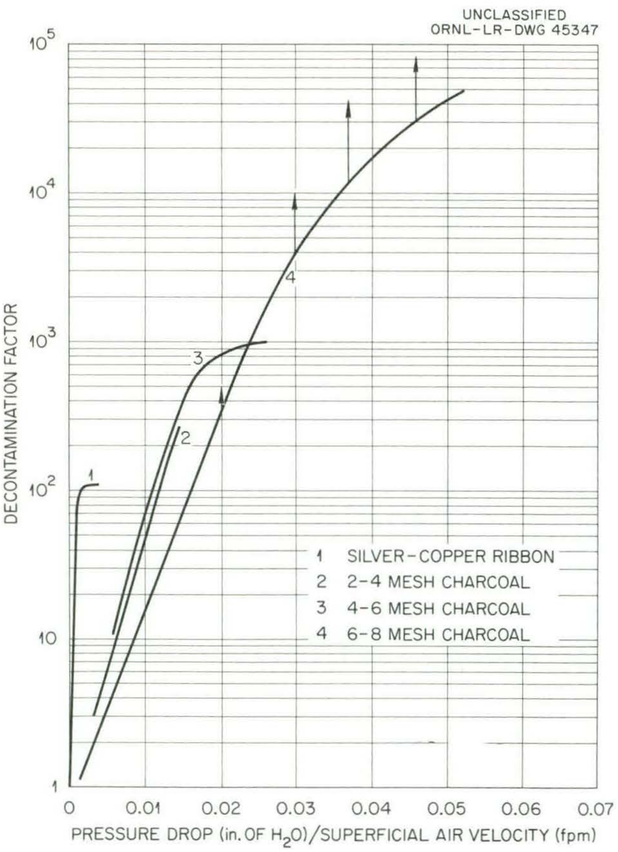

Fig. 7. Decontamination Factors as a Function of Pressure Drop and Superficial Air Velocity.

charcoal represents one interpretation for consolidating the data presented in Fig. 6 for this adsorbent. Although these curves are based on limited data, some tentative conclusions can be drawn regarding the relative merits of the silver-copper ribbon and several mesh sizes of charcoal for application at air velocities in the range 150 to 200 fpm. From the standpoint of pressure drop, silver-copper ribbon is superior to charcoal for decontamination factors up to 100. Between decontamination factors of 100 to 300, 2–4 or 4–6 mesh is more suitable; from 300 to 1000, 4–6 is the choice; above 1000, 6–8 mesh is preferable.

PRNC IODINE ADSORPTION SYSTEM

The quantity of iodine likely to be released from a swimming pool reactor core through an accident and the resulting air concentration are speculative matters. Laboratory experiments on the release of iodine vapor from melted reactor fuel elements have shown that the quantity depends upon the fuel type and the identity of the atmosphere, among other variables (*6*). The amount of iodine that would actually reach the charcoal is also open to question, since iodine vapor would be expected to plate out on most exposed surfaces in the building, based upon reported results of iodine deposition and air diffusion experiments (*5, 13*). In addition, a large portion of the iodine will be retained in the particle filter. Experiments are reported which indicate that CWS-type filters will remove 90% or more of the radioiodine contained in moist air streams (*10*). Therefore, air concentration of iodine could vary over wide limits, and, for this reason, activated charcoal would be preferable to silver-plated copper ribbon since charcoal has a high efficiency over wide concentration ranges.

The pressure drop through a charcoal mass can be reduced by decreasing the depth of adsorbent and increasing the area exposed to air flow. This constitutes reducing the superficial velocity of air flow through the charcoal. A commercial canister which contains the charcoal between two concentric cylinders having perforated walls, thus increasing the exposed charcoal area, is shown in Fig. 8. Canisters of this type are available from the Charles E. Manning Company, Pittsburgh, Pennsylvania, and the Connor Engineering Corporation, Danbury, Connecticut. Several canisters, designated as type H-42 and having a charcoal depth of 0.75 in., were purchased from the Connor Engineering Corporation and tested. Each canister, containing 1.5 lb of 6–14 mesh charcoal, is rated to process air at a maximum of 25 cfm and was tested for iodine vapor adsorption at that flow rate.

Three canisters were arranged in series, as shown in Fig. 9. The iodine vapor was injected

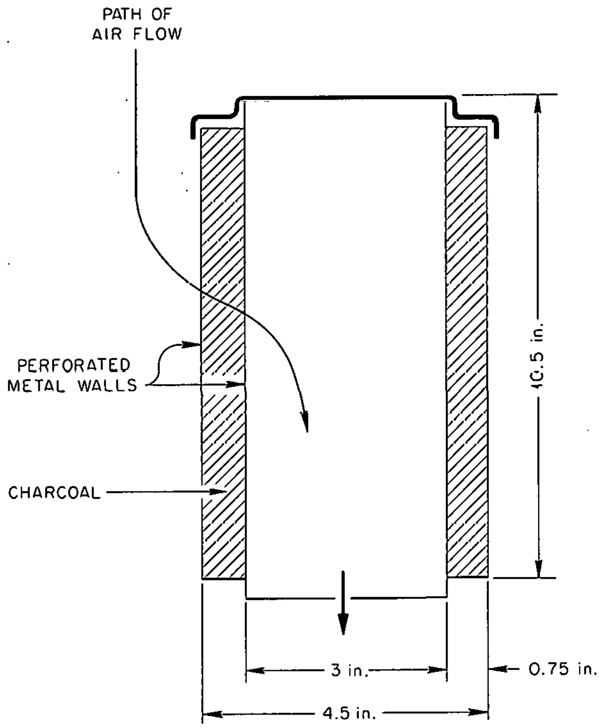

Fig. 8. Cross Section of Activated Charcoal Canister.

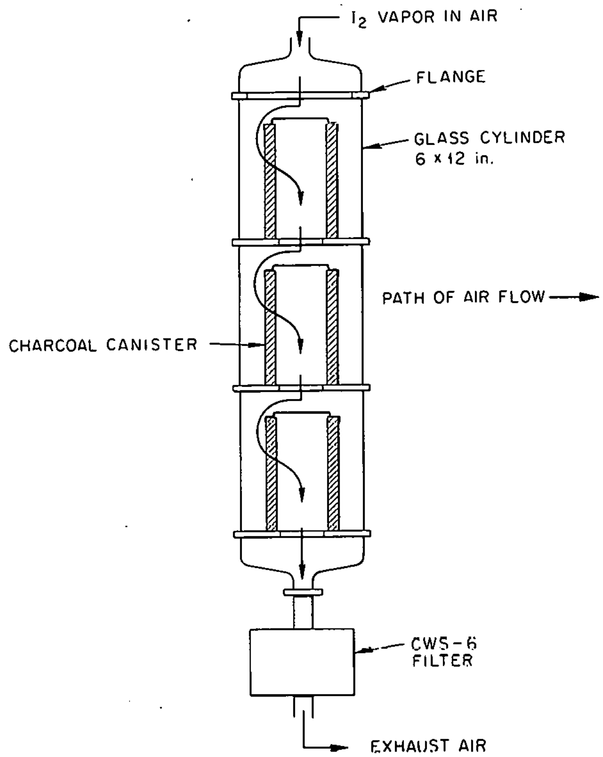

Fig. 9. Experimental System for Canister Test.

for 40 min, with an average concentration of 0.015 mg of I^{127} and 4 μc of I^{131} per cubic foot, and the air flow through the system was continued for 24 hr. Each canister has an exposed surface of 1 ft^2, and at a flow rate of 25 cfm, the superficial air velocity through the charcoal is 25 fpm. In addition to using the collector shown in Fig. 8, an attempt was made to detect iodine activity by monitoring a large volume of the exhaust air with a gamma-ray spectrometer, but the results were negative. The entire assembly was analyzed for I^{131} after completion of the test. The three-stage system had an efficiency of 99.998%, with the first, second, and third canisters exhibiting efficiencies of 99.99, 74.6, and 14.5%, respectively.

A small portion of the iodine-containing charcoal from the first canister was placed in a section of 1-in.-dia glass pipe, and air at a superficial velocity of 75 fpm was passed through the charcoal in an effort to sweep out the iodine. The iodine activity in the system was monitored periodically by a gamma-ray spectrometer. After 250 hr the activity was still decaying with a half life identical to that of I^{131} (8.05 days). This information, displayed in Fig. 10, supports the assumption that iodine is permanently adsorbed by the charcoal, or, in other words, reacts chemically with the charcoal surface and is not readily removed by continued air flow.

It is proposed that activated charcoal be used in the emergency exhaust system of the PRNC reactor building. Canisters, of the type tested in the laboratory, or equivalent, are suitable if one canister is used per 25 cfm of exhaust air. A large number of canisters may be installed in parallel by utilizing a patented manifold mounting plate available from the manufacturer of the canisters. Pressure drop through one H-42 canister at 25 cfm is 0.15 in. of water, as quoted in the manufacturer's literature.

The problem of heating in the charcoal and consequent oxidation is not considered to be serious in this application since the heat released by a reactor accident will be dissipated in the pool water and the large volume of air in the building. The iodine would be adsorbed in a large number of canisters, and the local heating of the charcoal by beta decay would be minimized by the cooling effect of the exhausting air. Control of combustion, if it should occur, could be handled by diverting the air flow to an alternate charcoal system and allowing the affected canisters to burn out. A smouldering canister would not be expected to ignite adjacent canisters, and only a small fraction of iodine contained in the system would be released. This iodine would deposit at other points, and the release to the atmosphere would be almost nil. Experimental tests of the control of charcoal fires have been reported (*2*). In addition to the particle filter upstream from the charcoal, it is suggested that an additional filter be installed downstream from the charcoal to prevent the discharge into the atmosphere of fine, iodine-laden dust particles which may be released from the charcoal mass.

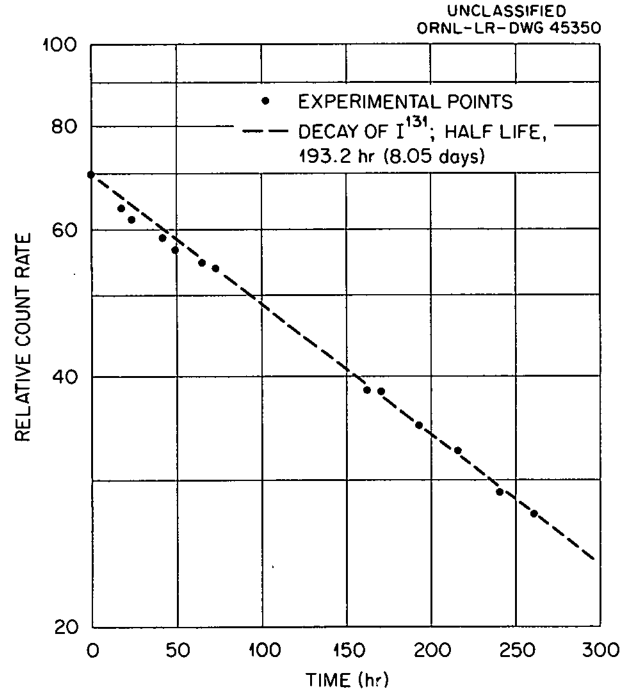

Fig. 10. Decay Rate of I^{131} in a Dynamic System.

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